APS Renewal: Small angle x-ray scattering (SAXS)

SAXS has been a crucial technique for a wide variety of materials and in situ studies. With the benefits of high flux and well collimated source like APS, it is getting significant attentions from many fields such as Chemistry and Catalysis, Condensed matter and Material physics, Applied Science, Geological/Environmental Science, Life Sciences, and Surface/Interface/Thin film. Especially in the field of nanoscience, SAXS has been so critical and popular as SEM or TEM. It has been a tool to measure shape/size/density of nanoscale (1nm ~ 1um) objects in any condition (solution, solid, gas, high/low pressure and so on). It has been the most accurate size measurement technique in nanoscience even without destroying samples. Recently SAXS has added chemical sensitivity (ASAXS) and surface/thin film probe (GISAXS).

In five years SAXS capability of APS will be upgraded significantly in terms of available beamtime as well as its performance. While two dedicated beamlines for SAXS-related-techniques, Grazing Incidence Small Angle X-ray Scattering (GISAXS) and X-ray Photon Correlation Spectroscopy (XPCS), have been recently launched and been successful, but SAXS in general has suffered from severe oversubscription. The first dedicated SAXS stations at APS, one of which will be in full operation at 12ID within two years and the other of which serve about 40% for SAXS, and dedicated 5ID SAXS beamline in 7-10 years, will address increasing demands for this technique. Nevertheless having two productive SAXS instruments, USAXS and current SAXS at 12ID-C, at a single line and letting them compete would starve users. Performance of SAXS is significantly affected by stability of beam, collimation, flux, high dynamic range and low background detectors, energy range/resolution, sample environment, and analysis software. High flux and collimation distinguish SAXS beamlines at APS from those at the other facilities. However the other criteria have limited the full use of SAXS capability at APS and the upgrades being planned will address these issues. A dedicated beamline would provide better collimation, more stable beam, and optimized sample environment. Also high performance detectors, such as PILATUS, will provide major breakthroughs for SAXS at APS due to high dynamic range, no dark/readout noise, and fast readout (less than 10m seconds) which are the most critical components for SAXS.

Upgrade plans throughout APS beamlines can be categorized as more beamtime, wider q range (better collimation, SAXS/WAXD, or fast setup change), faster acquisition (down to milliseconds by improved flux and detector), and simultaneous measurement (chemical probe adds). This will cover needs for both efficient routine measurements and extreme frontier science. SAXS group at APS is noting the trend of combining techniques, which can help to overcome limit of SAXS by adding chemical probe or imaging capabilities. An impediment for becoming a leading characterization and analytical facility in nanoscience is the lack of an overall plan for SAXS at APS. For instance, a suite of data reduction and analytical software tools that are common to all SAXS at APS will boost user productivity and promote theoretical studies. Shared sample preparation laboratories and support for engineering of experimental apparatus could be also considered. There are discussions on these issues among SAXS groups and hopefully some types of outcome in five years.

Summary of future capabilities is

Solution SAXS: Solution SAXS for biological samples will be the largest user community benefitted from high performance low noise detector. Fast (greater than 100 Hz) and extremely low background detectors will enable various real-time monitoring of biological phenomena at the nanoscale. Both SAXS beamlines, 12 and 18ID, will provide improved ASAXS capability that will enhance phase determination for solution SAXS. Significantly reduced sample volume, less than 20ul, will be required.

ASAXS (Anomalous SAXS): With a high resolution monochromator which will be available at 12ID and is designed for spectroscopic purpose, ASAXS could be done in the range of 7-40keV and EXAFS will be measured simultaneously. This upgrade will enable to study various high Z materials: Researches dealing with samples having multi-elements such as nanoparticle alloy, embedded particles, or composites will be benefitted. Chemical status of elements can be resolved after upgrade.

GISAXS: GISAXS has been of interest in self-assembly or growth of nanoparticles on or embedded in interfaces. Higher energy, up to 30~40 keV, will be available at 8ID and 12ID, which will allow studying nanoparticles/nanostructure formation under water and embedded under high Z materials. Tens of micro-second time resolution will be available with higher spatial resolution achieved by focused optics. In addition, GI-EXAFS and GISAXS may be carried out simultaneously at 12ID with mass-spectroscopy which is useful for in-situ catalysis studies, where for example GI-EXAFS can probe oxidation/reduction of nanoparticle catalysts and GI-SAXS determine their morphology at the same time.

USAXS (Ultra small angle x-ray scattering) will be served at 12ID with improved beam stability with faster scanning, 1-2 minutes (Currently 15min). 2D collimation will enable 2D USAXS, which is ideal to study anisotropic materials without any smearing. USAXS imaging will be available routinely.

XPCS : The upgrade plan for 8ID XPCS aims to expand its time scales from the slowest neutron spin echo measurements [$O(10^{-6}$s)] to near static [$O(10^{3}$ s)] conditions with at best 50 times of increased flux.

SAXS/WAXD: 5ID is planning dedicated beamline for simultaneous SAXS/WAXD in the 7-10 year vision. This capability with a PILATUS detector (no dark/readout noise and 3.8 ms readout time) would cover 0.002 to 2 $Å^{-1}$ seamlessly. High energy SAXS/WAXS also has been proposed at 1ID and 11ID, which will then provide inner atomic structure of nanoparticle from pair distribution function as well as shape/size from SAXS simultaneously.

High Energy SAXS: High energy SAXS has been available at 1ID and will be upgraded for faster timing (4 ms readout) with wider area of measurement (400*400 mm^2). In addition this capability is going to be added to 11-ID-B to operate in parallel with PDF measurements. This technique is essential to see embedded nanostructures such as void in high Z thick materials.

MicroSAXS: About 10um size x-ray beam for SAXS will be available at 12ID dedicated SAXS station with the help of a focusing optics. This will help to visualize locally located nanostructure such as self-assembly at interfaces or to better resolve individual cluster of nano-objects of less than 100um in size.

High throughput SAXS: 12ID-B dedicated station is planned to be high throughput SAXS beamline. It will have a detector in a vacuum flight tube, which will allow fast switch of sample-to-detector distance. Some portion of beamtime will be set aside for a rapid access.

Time-resolved SAXS: Down to tens of nanosecond time resolution could be reached with 12ID pink beam and fast detector such as annular Si-based detector. Tens of micro second time resolution will be available at all ID SAXS beamlines.